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Processing and Characterization of Welded Bonds Between Thermoset and Thermoplastic Composites

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Abstract

To assemble complex structures with short cycle times, the feasibility of welding thermoplastic (TP) to thermoset (TS) composites is demonstrated using a phenomenological approach. The effect of the thermal degradation of the TS composite (AS4/3501-6) on its shear strength is assessed in order to minimize the TS degradation during the welding process. The degradation is reported to decrease the shear strength in an exponential way. A layer of TP (polysulphone [PSU]) is cocured at the surface of the AS4/3501-6 laminate during its cure cycle in an autoclave. Two laminates are then welded together using a resistance welder. The highest shear strengths, which are close to the shear strength of bulk AS4/3501-6 autoclave-processed baseline, occur for a welding time and temperature of 10 s and 300 °C, respectively. Higher and lower temperatures induce degradation and low consolidation of the weld, respectively. The welding interface between the TP layers is the strongest, while failure is mixed and occurs at interfaces between TS and TP and at interfaces between TP and heating element at the fiber/matrix interfaces. Further investigations are required to optimize the welding processing parameters and to identify the bonding, degradation, and failure mechanisms using a more fundamental and molecular approach.

Table of Contents

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List of Figures

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List of Tables

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1. Introduction

This report studies the feasibility of a new technique whose advantages overcome the drawbacks of using mechanical fasteners or chemical adhesives for joining thermoset (TS) composites. Furthermore, this technique, called fusion bonding, allows two TS parts to be joined in shorter time, with the possibility of future disassembly of the complex bonded structures. This technique has already been applied to join thermoplastic (TP) composites, but can reasonably be applied to join TS when a layer of TP is cocured on the surfaces to be joined. Subsequently, two types of interfaces are then responsible for the strength of the final piece, that is to say, the TS/TP interface and the TP/TP joining interface.

1.1 Thermoset/Thermoplastic Bonds. The bonding between a TS and TP is dictated by the chemical compatibility of the two materials. The degree of chemical compatibility can be defined by a unique glass transition temperature (T_g) or by two similar solubility parameters (δ). Indeed, the square of the difference between the δ of the two materials is directly related to the enthalpy of mixing ΔH_m. In order to have a spontaneous mixing, the Gibbs free energy of mixing (ΔG_m) has to be minimized; this is reached by minimizing ΔH_m and occurs when the two δ are similar. Once a TS and TP are chemically compatible, they can form an interpenetrating polymer network (IPN) [1] in order to ensure a high enough bond strength. This occurs during the cure cycle of the TS, whose monomers diffuse into the molecular chains of the TP [2] and form a three-dimensional network or IPN.

In the case of good chemical compatibility between the TS and TP materials, the technique of the film cocuring is used most often [2] to bond the TP film on the TS part during its cure cycle in an autoclave. In the case of chemical incompatibility, a new technique was recently developed, termed diffusion-enhanced adhesion (DEA) [3], and consists of adding a second TP film (TP2) at the TS/TP interface, which is chemically compatible with the TS and has a T_g close to the one of the TP. A last technique, less commonly used, is the TP hybrid interlayer that does not require chemical compatibility because the bonding is purely mechanical. The TS is bonded

to a carbon fiber-based TP (CF/TP), whose face to be bonded contains only an empty fibrous network in which the TS will diffuse during the curing [4].

1.2 Thermoplastic/Thermoplastic Bonds. Bonds between two TPs are more easily achieved because of the same nature of the materials, which can be easily molten during fusion bonding. However, there are still some bonding mechanisms to consider in order to control the bond quality. First, the two materials to be bonded require some degree of intimate contact, which is defined as the percentage of area that is in contact with the two surfaces to be bonded. Then, in a process called healing, an interdiffusion of the chains takes place and forms an entanglement of the chains. This is the reason for the high strength of the bond, similar to an IPN in the case of TS/TP bonds. During the last step, the consolidation pressure is applied in order to reduce the voids and reach an optimum thickness of the bond, which is achieved by squeeze flow of the TP.

Some processes developed for bonding two TPs together, such as the thermabond method [5], have been used with techniques like press or resistance welding. In the case of TS/TP welding, whose process is described in Figure 1, the temperature has to be between the T_g and the melting temperature (T_m) of the TP, but below the temperature at which the TS degrades.

A schematic of the resistance-welding process is shown in Figure 1; two CF/TS parts containing a cocured layer of TP are welded using an interlayer heating element. An alternating current source is applied to the heating element through its ends. Due to the Joule effect, the heating element gets hot enough to allow the two parts' surfaces to melt and be welded. Additionally, pressure is applied to ensure a good contact and consolidation during the cooling stage.

1.3 Approach. A phenomenological approach was followed in this work, which contained three different steps. The first step was related to the materials (TS and TP), which had to be

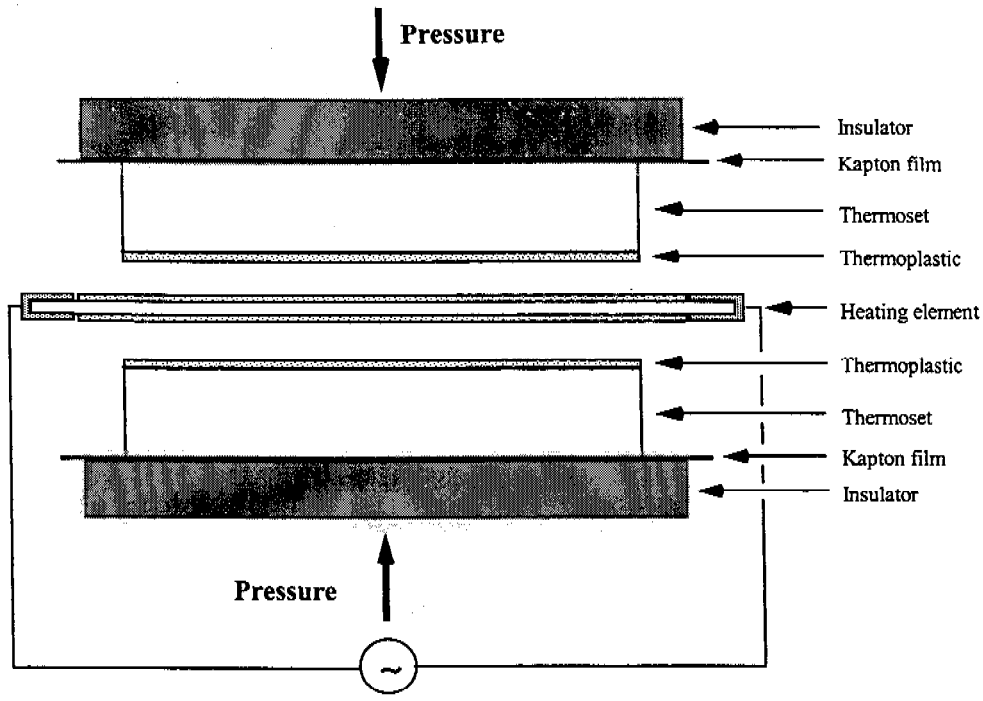

Figure 1. Resistance-Welding Process.

chosen in order to be chemically compatible to form an IPN. Furthermore, the TS needs to be sufficiently resistant to thermal degradation during welding. For this purpose, the effect of thermal degradation on mechanical properties of the TS was assessed first. The second step was processing the cocured TP layer on the TS part, followed by fusion bonding the two parts using a resistance-welding technique. The final step was the characterization of the welds by assessing the interfacial shear strength and the observation of the failure mechanisms.

2. Experimental

2.1 Materials. The TS used in this work was a unidirectional carbon fibers/epoxy (AS4/3501-6) from Magnamite Hercules, with a T_g of 215 °C. The TP was a polysulphone (PSU), with a T_g of 185 °C and a processing temperature between 300 °C and 360 °C. These materials have both good chemical compatibility and bonding quality [3], but AS4/3501-6 is also a commonly used TS for high-performance applications despite low thermal degradation

resistance. PSU has a high melt viscosity and a high temperature resistance. Furthermore, PSU has shown good welding properties in earlier applications [6].

2.2 Thermal Degradation. Thermal degradation of AS4/3501-6 is not yet well understood. In order to measure weight loss, several bulk AS4/3501-6 baseline specimens were degraded in a conventional oven, under vacuum, and at different temperatures and times going from 250 °C to 345 °C and from 10–60 min, respectively. The chosen temperatures and times were determined from isothermal thermogravimetric analysis (TGA) measurements, taking into account that, for the same temperature and time, the weight loss of a bulk specimen is lower than for a powder TGA. The degraded specimens were then tested, and the shear strengths were reported vs. the weight loss.

2.3 Processing. The bulk AS4/3501-6 baseline material and the AS4/3501-6 with the cocured PSU film were processed in an autoclave, following the cure cycle given by the AS4/3501-6 supplier. No enhanced cycle was used for the parts with the PSU film. The bulk AS4/3501-6 baselines were composed of 24 plies (0°). The cocured parts composed 24 plies of the AS4/3501-6 baseline and 1 ply of 0.15 mm or 0.30-mm-thick PSU film on one side of the laminate.

The heating elements for the resistance welding were vacuum bag processed in the oven and were composed of 1 ply of carbon fiber/polyarylsulphone (CF/PASU) sandwiched between two layers of 0.15-mm-thick PSU film. In order to ensure a good electrical contact between the fibers and the electrical circuit, metal meshes were then ultrasonically welded at both ends of the composite heating elements (Figure 1).

2.4 Welding. The controlled parameters for the welding process were essentially the power, pressure, and dwell time. The power was set to 50 kW/m^2 for all welds. The pressure was set to 0.38 MPa and 0.17 MPa for the parts with the 0.15-mm- and 0.30-mm-thick cocured PSU layer, respectively. The temperatures and dwell times were set from 250 °C to 300 °C and from 45 s to 300 s, respectively, and were chosen case by case,

depending on the quality and shear strength value of the preceding weld. For the first weld, the parameters were chosen according to previous work [6], where CF/PASU was used instead of AS4/3501-6.

2.5 Testing. Two test methods were used in order to assess the shear strength of the degraded bulk AS4/3501-6 composite as well as the weld strengths. The first one was the apparent interlaminar shear strength test (ILSS) [7], used for the degradation assessment. Since this test was not efficient enough for the testing of the welds, the interlaminar shear device test (ISD) [8] was used for this purpose. Normalized to the nondegraded bulk AS4/3501-6, these two test methods gave identical results. In the following sections, only the normalized shear strengths (NSS) are compared. Test samples were cut out from several positions of the welded samples.

3. Results and Discussion

3.1 Degradation. As shown in Figure 2, the thermal degradation of the bulk AS4/3501-6 baseline is evidenced and reported to decrease the shear strength vs. the weight loss in an exponential way. The legend shows at which temperature and time the specimens were degraded. The two corresponding photographs illustrate the effect of the thermal degradation, which manifests by a weight loss, on the microstructure of the specimens having no weight loss and 7.5% weight loss, respectively.

For degraded composite, delamination between the plies is responsible for the very low shear strength. For the case of no weight loss, the scatter in shear strengths in Figure 2, is certainly due to a lower initial consolidation between the plies because a loss of vacuum pressure occurred during the curing in autoclave. On the other hand, for the 7.5% weight loss, there is an evident loss of resin between the plies, which is certainly due to changes in the chemical structure of the resin.

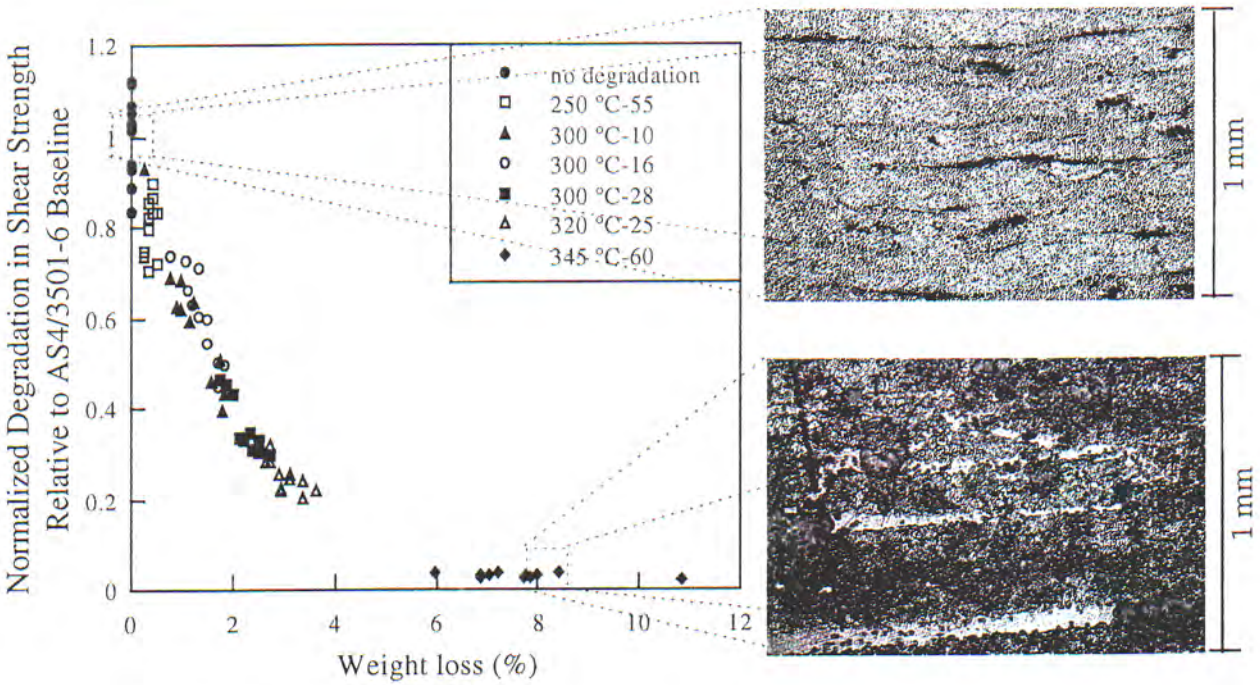

Figure 2. Effect of Thermal Degradation on the Bulk AS4/3501-6 Baselines.

It is also observed that the difference in terms of weight loss between TGA measurements and degradation in the oven decreases with exposure to increasing temperature. The measured bulk sample (oven) weight loss is consistently less than that determined by TGA measurements (Table 1).

Table 1. Differences of Weight Losses Between TGA and Degradation in an Oven for Several Exposure Conditions.

Temperature (°C)	Time (min)	TGA Weight Loss (%)	Oven Weight Loss (%)	Deviation (%)
250	55	1.0	0.39	61.0
300	10	1.5	1.20	20.0
300	16	2.0	1.37	31.5
300	28	3.0	2.28	24.0
320	25	5.0	3.05	39.0
345	60	8.0	7.69	3.9

Applying these results to the welding process, the thermal degradation of the AS4/3501-6 is expected to be negligible (2% or less) if welding occurs around 300 °C for a few minutes.

3.2 Thermoset/Thermoplastic Bond Quality. The quality of the bonds between the AS4/3501-6 and the PSU film is reported to be good because some 3501-6 resin diffused and even flowed through the whole thickness of the PSU film during autoclave cure. These parts are not used for welding because the surface to be welded is not pure PSU but a blend of both PSU and 3501-6 resin, which could alter the quality of the welded bond. In order to control an efficient diffusion of the 3501-6 monomers, an optimization of the cure cycle has to be studied.

3.3 Welding Properties. After several experiments to optimize the welding parameters, it is observed that the quality of the heating element and the contact of the welded metal meshes with the prepreg are very important in order to ensure reproducibility. The polymer flow at the edges can be reduced by lowering the pressure, and the local overheating can be minimized by adding two cooling nitrogen fluxes on the edges along the fiber direction.

Although the control of the welding process is not optimum and the reproducibility is not ideal, three different results are experienced. The first case is related to the welds with the highest NSS, which is obtained at a process temperature of 300 °C for 10 s (Figure 3). The average NSS is homogeneous in the plane of the weld interface, and the NSS is close to the bulk AS4/3501-6 shear strength (NSS = 1). It can be concluded that the interdiffusion of the PSU chains and the formation of IPN occurred with these welding parameters.

The second case is observed for welds that have an average NSS near 0.5 (Figure 4) and that are processed at 250 °C for 300 s. Again, the NSS is homogeneous in the plane of the weld interface; however, the shear strength is half that of the bulk AS4/3501-6. This is evidently not due to thermal degradation but to a low bond consolidation and incomplete interdiffusion of PSU at this lower temperature.

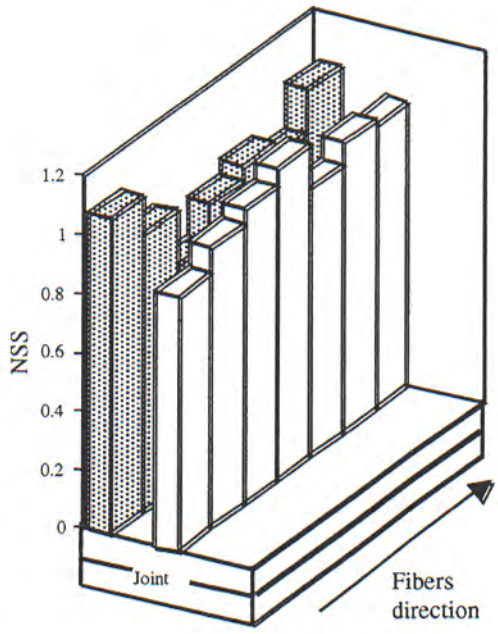

Figure 3. Results of Two Typical Good-Quality Welds With a Shear Strength Close to the Bulk AS4/3501-6 Baseline. Bond Strengths Are Uniformly Distributed Across the Width of All Welded Areas.

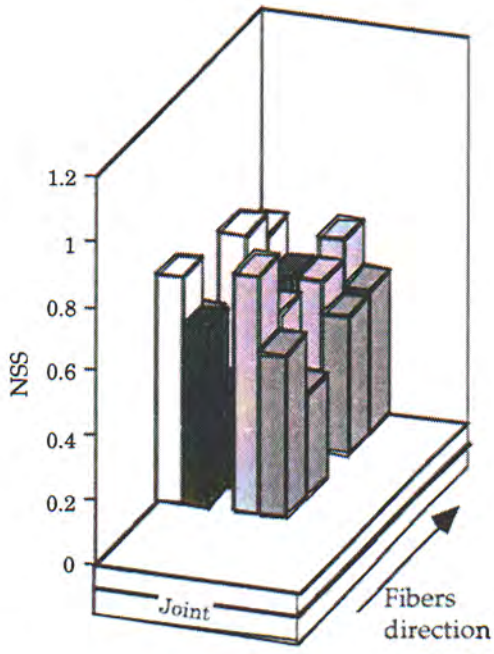

Figure 4. Results of Four Typical Poor-Quality Welds at Low Consolidation Temperature.

The third result represents the effect of the heat gradient across the weld. The edge along the fibers direction went up to 600 °C for 10 s (Figure 5), and the rest of the weld went to between 300 °C and 400 °C for 30 s. The NSS at the edge is low (due to degradation). The other NSS values are lower than unity. This is linked to a temperature above 300 °C for more than 10 s, which induces degradation. However, the locations of the highest NSS in Figure 5 are reported for temperatures around 300 °C. The three different behaviors (Figures 3–5) correlate quantitatively with the degradation results presented in Figure 2.

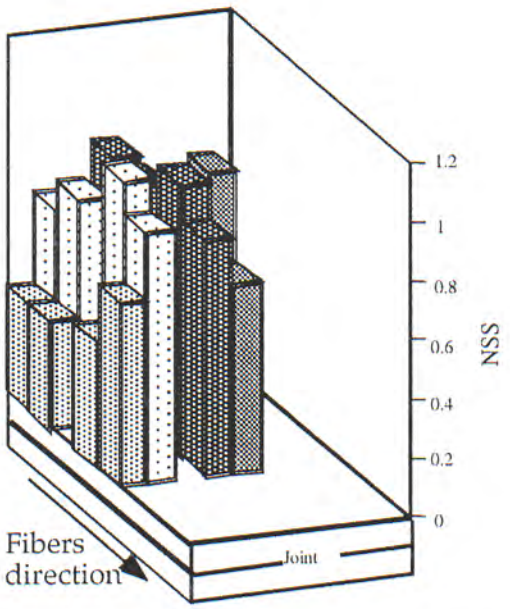

Figure 5. Results of Four Typical Welds Evidencing the Inhomogeneity of Thermal History Influencing the Distribution of the Bond Strengths.

Micrographs of the weld cross sections are presented as qualitative evidence of the degradation. As shown in Figures 6 and 7, the bond quality and, therefore, the NSS are completely different for a nondegraded and a degraded weld. The nondegraded weld contains no voids. The degraded weld shows degraded material and a high percentage of voids in regions close to the edges of the weld where the flow of the PSU is higher.

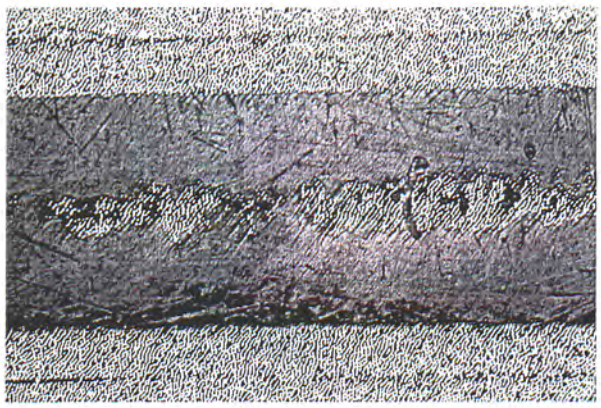

Figure 6. Good-Quality Weld (NSS = 1).

Figure 7. Degraded Weld (NSS = 0.5).

3.4 Failure Mechanisms. In most of the cases, the failure of the welds is cohesive within the composite elements of the complex joint structure. As shown in Figure 8, the failure propagates at the interface between AS4/3501-6 and PSU (TS/TP), crosses the PSU layers, and propagates at the interface between the PASU prepreg of the heating element (TP/h.e.) and the PSU. The two failure interfaces (top and bottom) are of a different nature. The top interface is the result of two mechanisms. First, an adhesive rupture around the fibers is evidenced by the cleanness of some of the fibers, and then a plastic deformation of the PSU is shown by the rough and ductile surface aspect of the PSU. The bottom interface is produced only by an adhesive rupture around the fibers.

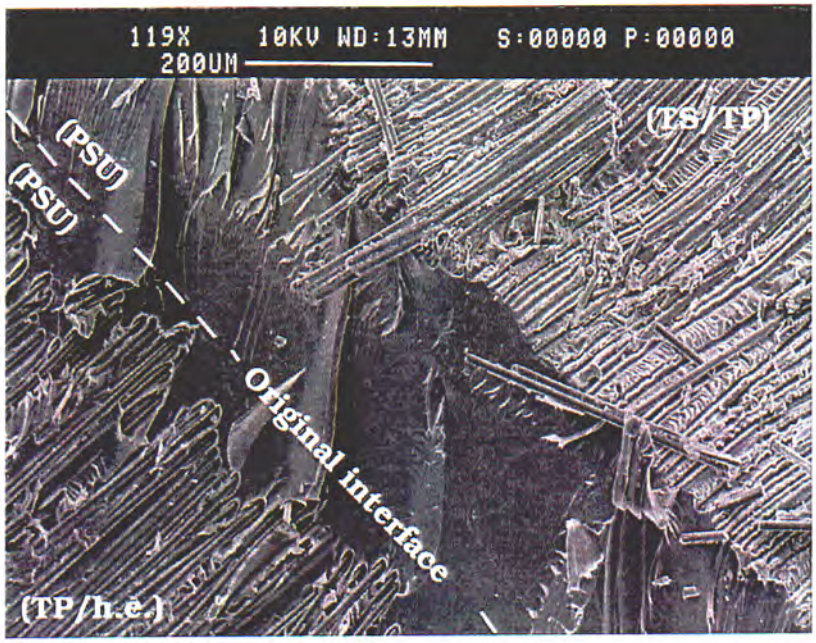

Figure 8. Typical Mixed Failure of the Weld.

These results indicate a competition between the failures of the two interfaces. The bottom interface is weaker because less energy can be absorbed by plastic deformation. No failure is observed at the original PSU/PSU interface. These interfaces are not even visible in Figure 8 (dashed line), which indicates a good interdiffusion between the thermoplastics.

4. Conclusion

The feasibility of welding TP to TS is demonstrated in this work. The thermal degradation is shown to affect the shear properties of the TS by decreasing its shear strength exponentially. During welding, degradation is negligible if correct welding processing parameters are used. The diffusion of the 3501-6 resin into the cocured PSU layer is obtained without the use of an enhanced diffusion cycle. Nevertheless, the conditions need to be optimized in order to better control the diffusion length.

The selected welding parameters resulted in three different scenarios. The bonds with the higher shear strengths are processed at 300 °C for 10 s. Lower temperatures induce insufficient

molecular interdiffusion and weak consolidation. Last, excessive temperatures induce degradation. The failure of the welds is dominated more by composite interfaces (fibers/matrices) than by the interfaces between thermoplastic and thermoset polymers. Bonding of dissimilar materials such as thermoplastics and thermosets holds promise for assembly of complex composite structures.

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To assemble complex structures with short cycle times, the feasibility of welding thermoplastic (TP) to thermoset (TS) composites is demonstrated using a phenomenological approach. The effect of the thermal degradation of the TS composite (AS4/3501-6) on its shear strength is assessed in order to minimize the TS degradation during the welding process. The degradation is reported to decrease the shear strength in an exponential way. A layer of TP (polysulphone [PSU]) is cocured at the surface of the AS4/3501-6 laminate during its cure cycle in an autoclave. Two laminates are then welded together using a resistance welder. The highest shear strengths, which are close to the shear strength of bulk AS4/3501-6 autoclave-processed baseline, occur for a welding time and temperature of 10 s and 300 °C, respectively. Higher and lower temperatures induce degradation and low consolidation of the weld, respectively. The welding interface between the TP layers is the strongest, while failure is mixed and occurs at interfaces between TS and TP and at interfaces between TP and heating element at the fiber/matrix interfaces. Further investigations are required to optimize the welding processing parameters and to identify the bonding, degradation, and failure mechanisms using a more fundamental and molecular approach.

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